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ASD TR 7-782(VII)

ASD Interim Report 7-782(VII)
February 1962

THE DEVELOPMENT OF OPTIMUM MANUFACTURING METHODS FOR COLUMBIUM ALLOY FORGINGS

P. F. Darby

CRUCIBLE STEEL COMPANY OF AMERICA
Midland Research Laboratory

Contract No. AF 33(600)-39944
ASD Project 7-782

Interim Technical Engineering Report
15 November 1961 - 15 February 1962

Sixteen Cb74 (10W-4Zr) closed die forgings were successfully made from 16 forging blanks using conventional forging equipment and techniques. No perceptible die wear resulted with a 2400F forging temperature whereas considerable die wear occurred when a 2200F forging temperature was used. Excellent oxidation protection was provided by the Al-Cr-Si coating.

BASIC INDUSTRY BRANCH
MANUFACTURING TECHNOLOGY LABORATORY
ASD Aeronautical Systems Division
United States Air Force
Wright-Patterson Air Force Base, Ohio

ABSTRACT - SUMMARY
Interim Technical Progress Report

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Open die side forging of extruded and annealed Cb74 to a smaller round produced center bursts. This defect was not encountered when the stock was swaged or flattened.

The high temperature tensile and stress rupture strengths of the 4% zirconium closed die forgings was equivalent to that previously reported for 6% zirconium Cb74 upsets. Room temperature and -40F tensile ductility was excellent in the longitudinal direction but the transverse room temperature ductility was low.

Oxidation resistance of the 4% zirconium Cb74 was the same as the 6% zirconium alloy in the 2000-2400F range but was superior in the 1500-1832F range.

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FOREWORD

This Interim Technical Progress Report covers the work performed under Contract AF 33(600)-39944, "The Development of Optimum Manufacturing Methods for Columbium Alloy Forgings" from November 15, 1961 to February 15, 1962. This includes completion of Item 8 of the contract.

The work performed under Contract AF 33(600)-39942, "The Development of Optimum Manufacturing Methods for Columbium Alloy Sheet" from August 1, 1961 to December 15, 1961 (Ref. AMC Interim Report 7-784(VI)) covering the final results of the evaluation of F48 and D31 ingot integrity is also reported herein (Appendix A).

The report is published for technical information only and does not necessarily represent the recommendations, conclusions or approval of the Air Force.

This contract with Crucible Steel Company of America was initiated under AMC Manufacturing Methods Project 7-782, "The Development of Optimum Manufacturing Methods for Columbium Alloy Forgings." It is administered under the direction of Mr. A. J. Merkle of the Basic Industry Branch, Manufacturing Technical Laboratory, Aeronautical Systems Division, Wright-Patterson Air Force Base, Ohio.

Mr. R. O. Carson of Crucible's Midland Research Laboratory is the engineer in charge and Dr. P. F. Darby, Supervisor of Melting Research and Mechanical Metallurgy Sections, is the Project Director. Mr. J. B. Guernsey is the engineer in charge of the F48 and D31 ingot integrity study.

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PUBLICATION REVIEW

Prepared by R O Carson
R. O. Carson
Staff Metallurgist

Approved by Paul Darby
P. F. Darby, Supervisor
Melting Research and Mechanical Metallurgy Sections

Approved by Howard Clark
H. T. Clark, Manager
Midland Research Laboratory

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INTRODUCTION

This is the seventh interim report under Contract AF 33(600)-39944 entitled "The Development of Optimum Manufacturing Methods for Columbium Alloy Forgings." This report was prepared by the Midland Research Laboratory of the Crucible Steel Company of America. The contract is being administered under the direction of the Manufacturing Technology Laboratory of ASD Aeronautical Systems Division, Wright-Patterson Air Force Base. Mr. A. J. Merkle is the Project Engineer.

The objective of this investigation is to determine the optimum manufacturing methods for the production of columbium alloy forgings. The project is divided into several items:

Items 1 and 2	- State of the Art Survey and Selection of Four Alloys. This has been completed and a report has been issued. (Interim I)
Item 3	- Comparison of Four Ingot Consolidation Techniques. This has been completed and a report has been issued. (Interim II)
Item 4	- Melt Ingots for Forging Studies and Evaluate. This has been completed and a report has been issued. (Interim IV)
Item 5	- Submit a forging test program (letter to Gabriel Campbell, WPAFB, September 30, 1960).
Items 6 and 7	- Forgeability Tests on Four Alloys: F48 (15W-5Mo-1Zr), D41 (20W-10Ti-6Mo), D31 (10Ti-10Mo), and Cb74 (10W-5Zr). (This is covered in Interim V.)
Item 8	- Development of Statistically Satisfactory Process for the Production of Closed Die Forgings. (Interims VI and VII)
Item 9	- Reproducibility Study on the Best Forging Process Developed Under Item 8.

Interim report VI described the manufacture of ingots and breakdown by extrusion required to produce forging stock for Item 8 of this forging contract. This report (Interim VII) covers the closed die forging and the evaluation of the forgings and forging technique completing the work required in Item 8.

TECHNICAL DATA

1.0 Side Forging Experiments

Three extruded pieces (1.620" diameter x 2" long) of 6% zirconium Cb74 from heat 98610 were annealed 1 hour at 2600F. After conditioning, the pieces were heated to 2450F and hammer forged on open dies. Light blows were used and the material was reheated when the temperature dropped to about 1500F.

A center burst developed in the first specimen after it had been reduced to 1-3/8" diameter.

The second specimen was flattened to 1-1/8" thick, at which time it was in good condition, then turned 90°. After a couple of blows in this direction it also center burst with the burst 1-1/4" long and extending completely through the 2" length.

The third specimen was successfully swaged to about 1-1/8" diameter. The ends of the bar were concave indicating greater working of the outside fibres than the center. Although the swaging was successful, it did not improve the transverse ductility after outgassing. A transverse room temperature tensile test specimen from the swaged bar fractured before the yield point was reached.

These results indicate that extruded and annealed Cb74 can be open die forged in one direction on its side or swaged but that it is subject to center bursting when it is open die forged to a smaller diameter.

2.0 Closed Die Forging

A drawing of the forging produced is shown in Figure 1. The six coated Al-10Cr-2Si forging blanks (1½" diameter x 5½" long) were placed two at a time in the furnace and held about 5 minutes at temperature. Attempts to forge a tong hold on the end of the first blank (719-1) to aid manipulation in the edging impression were unsuccessful because the surface of the piece forged out forming a pipe and cracking resulted. The failure to forge a tong hold eliminated use of the proper edger and therefore only the blocker and finish impressions were used.

The remaining 15 pieces were forged without difficulty. Duplicate forgings were made for each of the eight different combination of conditions evaluated and shown below:

Furnace Temperatures °F	Die Temperatures °F	Lubricants
2200, 2400	400, 600	Aquadag & Molycote G

3.0 Nondestructive Examination

After forging the pieces were grit blasted to remove the Al-Cr-Si coating. Ultrasonic examination indicated that all forgings were sound. The flash which was as thin as 0.05" was also free of defects. Zyglo inspection revealed minor surface defects in 4 of the 16 pieces. Metallographic examination of sections through these defects revealed laps extending from 0.001 to 0.015" in depth.

Because the edging impression was not used the end lugs did not completely fill and there was more than the usual amount of flash producing forgings which were oversize by about 1/16" (Figure 2).

The surface on the ends of several of the forgings contained scale pits resulting from the hard oxidation protective aluminum coating being pounded into the surface. The entire flash surface, where the coating had been removed by the forging operation had an excellent appearance.

The molten aluminum dip and diffusion technique used to coat these pieces, left some areas near the end of the forging blank that had coating up to 0.015" thick, whereas most of the remainder of the piece had about 0.003" coating. The rough surface on one end of forging 719-4 (Figure 2) was not contaminated and probably resulted from too thick a coating on this end. We expect to improve the surface of the finished forging in Item 9 of this contract through the use of a thinner coating which will be obtained by spraying. No perceptible die wear resulted after forging the eight pieces at 2400F. Considerable die wear was observed after forging 8 pieces at 2200F. The die which was designed and used for forging steel was made from SAE 4350 heat treated to 385 Brinell.

Forging condition details and the results of the post forging visual examination are given in Table I.

4.0 Macroscopic Examination

Several forgings were sectioned for examination of structure and for mechanical, oxidation and recrystallization tests.

Longitudinal and transverse sections of as forged and forged and annealed material are shown in Figures 4 and 5 respectively. The structure is uniform and fine grained.

5.0 Hardness

Vickers (10 Kg) hardness surveys were made on longitudinal and transverse sections of as forged and, forged and annealed material (Figure 6). The hardness in both conditions is uniform with the annealed hardness (average 225) only slightly lower than the as forged hardness (average 234).

6.0 Recrystallization

Transverse specimens (about 1/4" cubes) were removed from the end and center of forging 719-1 and exposed after being suitably protected from atmospheric contamination for 1 hour at temperature. The results in Table III indicate that the recrystallization temperature (defined by MAB as at least 50% recrystallization and a drop in hardness of at least 2/3 of the total drop from as rolled to fully annealed) is 2300F for the middle section of the forging and less than 2200F for the end lugs.

7.0 Oxidation

Ground specimens about 1/4" thick cut from the annealed forgings were used for oxidation tests. The results are shown in Table V and Figure 7. The oxidation of the 4% zirconium Cb74 tested here was similar to that reported in Interim Report V for the 6% zirconium Cb74 in the 2000-2400F range. At lower temperatures 1500-1832F the 4% zirconium alloy has considerably greater scaling resistance and about the same resistance to shell formation as the 6% material. The ignition temperature of the 4% zirconium is apparently higher than the 6% material.

8.0 Microstructure

The microstructure of the annealed forgings was uniform and fine grained (ASTM 7-9) with a fine uniformly dispersed precipitate (Figure 8). For the most part the surface of the as forged pieces was not contaminated. A few isolated spots with up to 0.004" contamination were observed. After heat treating using a suitable protective coating about

0.001" of surface contamination was visible. A few spots on the annealed forgings were contaminated to a depth of 0.009" indicating the necessity for a more thorough conditioning (pickling) to remove about 0.005" after forging and before annealing.

9.0 Chemical Analysis

Chemical analysis was made on a specimen taken from the center of forging 719-1. The tungsten, zirconium and carbon results shown in Table IV are in good agreement with the ingot analysis indicating a homogeneous ingot in respect to these elements. Oxygen and nitrogen contents of the forging are slightly higher than those reported for the ingot. It is believed that this is not due to contamination during processing, which would be expected to increase oxygen more in proportion to nitrogen than is the case here, but to slight segregation of these elements in the as cast ingot.

Hydrogen content increased from about 1 ppm in the ingot to 19 after forging and 37 after annealing. Hydrogen pickup during processing and the detrimental effect of hydrogen contents as low as 15 ppm on the room temperature ductility of columbium alloys was reported in Interim Reports V and VI. Fortunately the hydrogen content of these forgings was readily reduced to 4 ppm by a 2 hour vacuum treatment at 1500F.

10.0 Tensile Tests

A transverse room temperature tensile specimen from the annealed extrusion made from ingot 98719 had very low ductility similar to that reported in Interim Report VI for the 6% zirconium Cb74.

Tensile ductility of the forgings in the longitudinal direction was excellent at room temperature and -40F. Room temperature transverse tensile ductility was very low in the center round section which had received relatively little working during the closed die forging. Good transverse tensile ductility was obtained in the heavily worked end lugs.

The 2000-2600F tensile test results obtained from longitudinal specimens taken from the middle portion of the forgings are in good agreement with those reported in Interim Report V for the 6% zirconium Cb74. The strength dropped fairly rapidly as the temperature was increased from room temperature to 1000F, went through a plateau or

maximum from 1000 to 1500F and again dropped above 1500F to 2600F. The ductility was good at all temperatures although a drop to 20% elongation occurred at 1500F. This increase in strength and decrease in ductility in this region is believed to result from strain aging.

All specimens were pulled at a rate of 0.05"/inch/minute cross head speed from zero strain to fracture. Vacuums were less than 1 x $10^{-[illegible]}$ mm Hg except for short periods during heat up when the pressure rose to as high as 5 x 10^{-4} mm Hg. Hot leak rate was usually about 10 microns an hour.

Microscopic examination of cross sections from the specimens showed many of the specimens were free from surface contamination. The 2600F specimen which was the worst had only 0.001" on the surface that was slightly contaminated. In no instance was there any increase in microhardness at the surface. It is believed that the very slight surface contamination detected on a few specimens would not significantly affect the results.

11.0 Stress Rupture Tests

Stress rupture tests were made on specimens taken from forging which had been annealed 1 hour at 2400F. Stresses were selected to give lives of about 10 minutes, 1 hour and 10 hours at temperatures of 2000, 2200, 2400 and 2600F. Vacuums were always less than 10^{-4} mm Hg and usually in the low 10^{-5} or 10^{-6} range.

The stress rupture strengths given in Table VII and Figure 10 are about the same or perhaps slightly higher than those reported in Interim Report V for the 6% zirconium Cb74 upsets. The 2400F 13,000 psi result was not used in plotting Figure 10 as it appears to be in error.

Microscopic examination indicated slight contamination on most of the specimens (Table VII). This was generally less than 0.001" and probably would not significantly affect the results.

TABLE I

Forging Conditions

Order Forged	Specimen Number	Furnace Temperature °F	Finish Temperature °F	Die Temperature °F	Die Lubricant	Appearance of Forgings
1	719-1	2400	1570	400	Aquadag	Satisfactory surface. Attempt to forge tong hold not successful. Reheated and forged in blocking and finishing impressions.
2	713-3	2400	1620	400	Aquadag	Good surface.
4	711-2	2400	1475	400	Molycote G	Good surface.
5	720-4	2400	~1600	400	Molycote G	Good surface
6	713-4	2400	~1500	600	Molycote G	Rough surface on left end both sides.
7	719-2	2400	~1620	600	Molycote G	Rough surface on right end one side. Two small Zyglo indications.
8	720-3	2400	1640	600	Aquadag	Rough surface on 1/3 of forging right end both sides. Surface on left end on one side is checked and file hard indicating contamination. Al-Cr-Si coating did not adhere on one end of this piece so it was patched with a non-metallic coating. Apparently this coating did not prevent contamination. Remainder of pieces were free of surface checking and surfaces were file soft.

TABLE I
(Continued)

Order Forged	Specimen Number	Furnace Temperature °F	Finish Temperature °F	Die Temperature °F	Die Lubricant	Appearance of Forgings
9	711-1	2400	-	600	Aquadag	Rough surface left end. One small Zyglo indication.
3	711-3	2200	1590	400	Aquadag	Good surface. Two small Zyglo indications.
16	720-1	2200	1575	400	Aquadag	Good surface except for large diameter left end.
14	713-2	2200	1580	400	Molycote G	Good surface except for small area at left end.
15	719-4	2200	1570	400	Molycote G	Bad surface on right end. Believe coating was too thick on this end.
10	713-1	2200	1605	600	Aquadag	Good surface. Small Zyglo indication at lug runout.
11	711-4	2200	1590	600	Aquadag	Good surface. Slightly rough at one end.
12	719-3	2200	1600	600	Molycote G	Surface rough at one end.
13	720-2	2200	1605	600	Molycote G	Good surface except for large diameter at left end.

TABLE II

Recrystallization of Forged Cb74

Transverse Section From Middle of Forging 719-1

Temperature	% Recrystallization	ASTM Grain Size	Hardness VPN (10 Kg)
As forged	0	-	235
1 Hr @ 2200F	40	-	228
1 Hr @ 2300F	70	7	219
1 Hr @ 2400F	95	7	214
1 Hr @ 2500F	100	6	213

Transverse Section From End Lug 719-1

Temperature	% Recrystallization	ASTM Grain Size	Hardness VPN (10 Kg)
As forged	0	-	259
1 Hr @ 2200F	100	8	217
1 Hr @ 2300F	100	9	221
1 Hr @ 2400F	100	9½	223
1 Hr @ 2500F	100	8	217

TABLE III

Chemical Analysis

Identification	Tungsten	Zirconium	Carbon	Oxygen	Nitrogen	Hydrogen ppm
Forging 719-1 (As Forged) (Heat 98719)	9.94	4.07	.01	.033	.016	19
Ingot Analysis - 98719	9.84	3.94	.01	.017	.010	1.5
Forging 719-4 (Annealed 1 Hour at 2400F)						37
Forging 719-4 (Annealed 1 Hour at 2400F + 2 Hours in Vacuum at 1500F)						4

TABLE IV

Cb74 Oxidation Test Results

Temperature °F	Time Hours	Shell Mils/Side	Scale Mils/Side
1500	10	6	7
1832	10	24	7
2000	10	39	5
2200	10	67	7
2400	10 minutes	46	2
2400	1	75	3
2400	10	90	12
2600	10	100+	51

TABLE V

Longitudinal Tensile Properties of Cb74
(Annealed 1 Hour at 2400F)

Specimen	Test Temperature ^{o}F	Ultimate Tensile Strength psix10^{-3}	0.2% Yield Strength psix10^{-3}	% Elongation	% Reduction in Area
711-2-E4	-40	98.1	83.6	38	69
711-2-E1	RT	85.0	75.0	30	78
713-1-M2	500	75.6	74.0	32	83
713-1-M3	1000	59.1	40.1	31	87
713-1-M4	1500	65.1	46.0	20	79
713-1-M5	2000	51.8	44.9	36	70
713-1-M6	2200	43.5	41.8	45	68
713-1-M7	2400	30.8	26.4	68	95
713-1-M8	2600	23.9	23.1	80	98

Note: All specimens were vacuum annealed ½ hour at 1400F.

TABLE VI

Room Temperature Transverse Tensile Properties

Specimen[1]	Ultimate Tensile Strength psix10^{-3}	0.2% Yield Strength psix10^{-3}	% Reduction in Area	Specimen Location
720-2-M2	87.6	83.8	1	Middle of forging perpendicular to flash.
720-2-M1	91.3	-	7	Middle of forging and parallel to flash.
720-2-E5	98.6	86.5	24	End lug.
720-2-M3[2]	97.8	87.2	56	

(1) All specimens were substandard size, ½" overall length with test portion 0.200" diameter and about 3/16" long.

(2) Longitudinal specimen.

TABLE VII

Stress Rupture Properties of Cb74
(Annealed 1 Hour at 2400F)

Specimen	Test Temperature °F	Stress psix10^{-3}	Rupture Life Hours	Elongation %	% Reduction in Area	Depth of Contamination in Mils as Revealed by Microscopic Examination
713-2-E1	2000	47	0.3	34	71	0.3
713-2-E2	2000	36	2.8	37	68	0.3
713-2-E3	2000	30	13.8	66	85	0.5
713-2-E4	2200	32	0.5	44	76	0.2
713-2-M1	2200	24	2.9	75	80	1.0
713-2-M2	2200	19	12.1	82	88	
713-2-M3	2400	24	0.25	70	82	0.0
713-2-M4	2400	15	4.3	85	85	1.0
711-3-E1	2400	13	2.5	100	91	
711-3-E2	2600	20	0.3	68	86	0.3
711-3-E3	2600	10	3.0	119	91	1.0

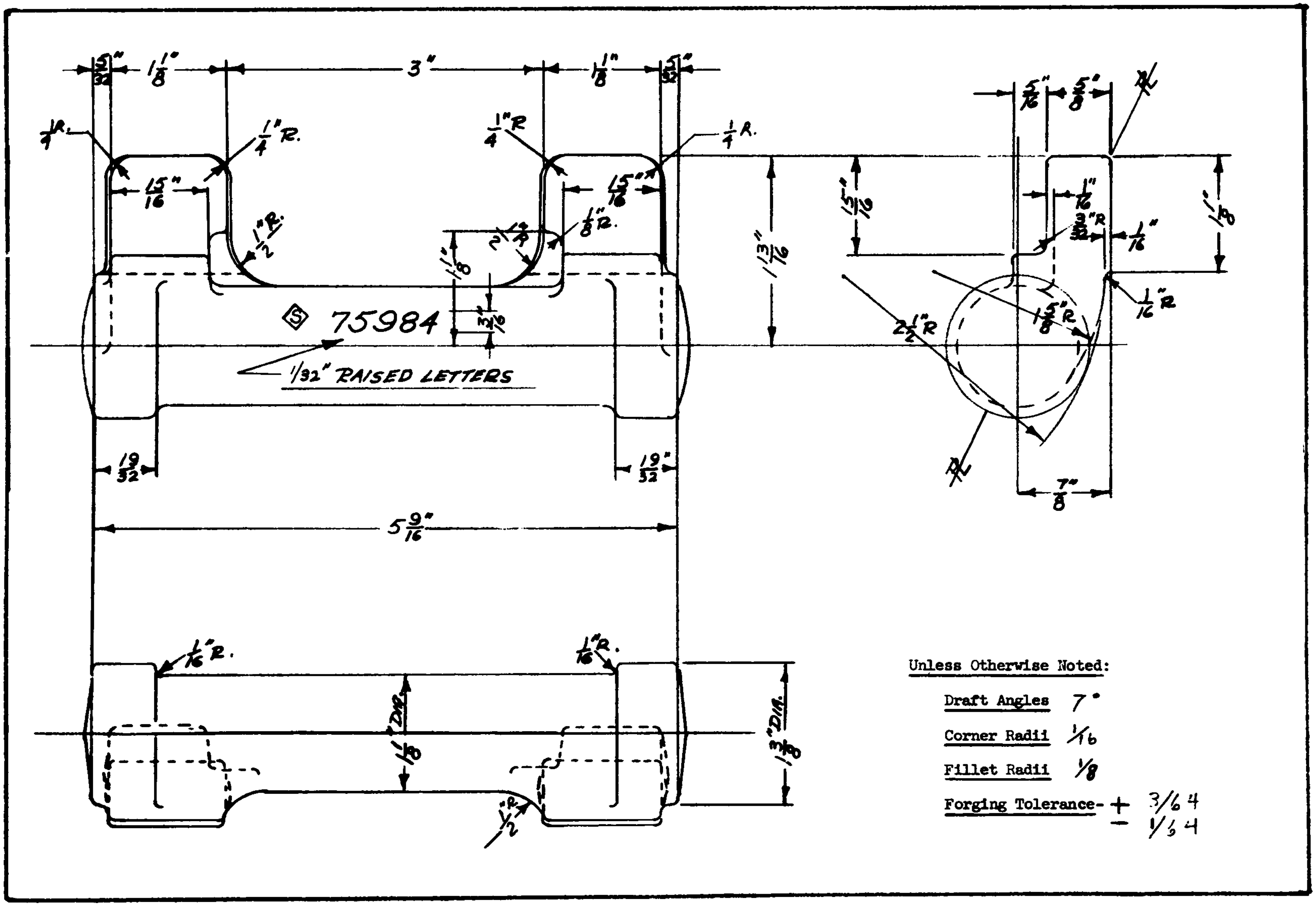


Figure 1: Closed Die Forging

Figure 2: Cb74 Closed Die Forgings 711-2, 720-2 and 719-4 in As Forged Condition

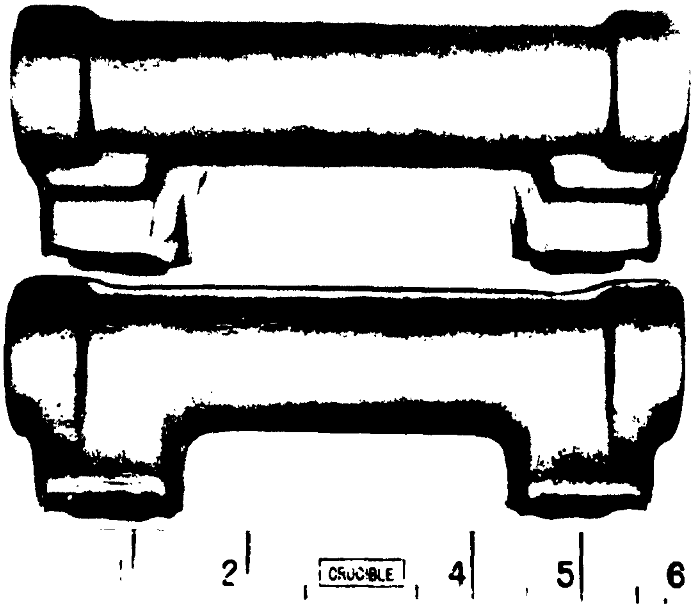


Figure 3: Closed Die Forgings 713-3 and 713-4 After Removal of Flash

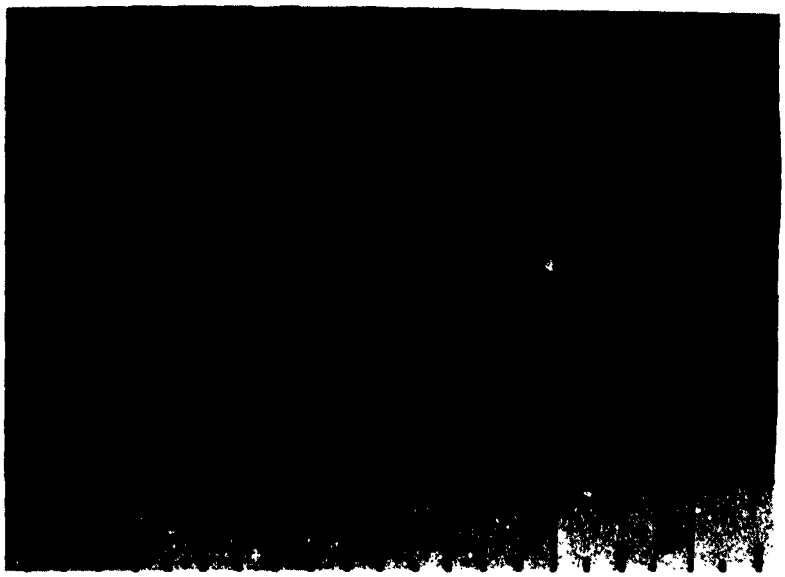


Figure 4: Macrostructure of Longitudinal and Transverse Sections of As Forged Cb74

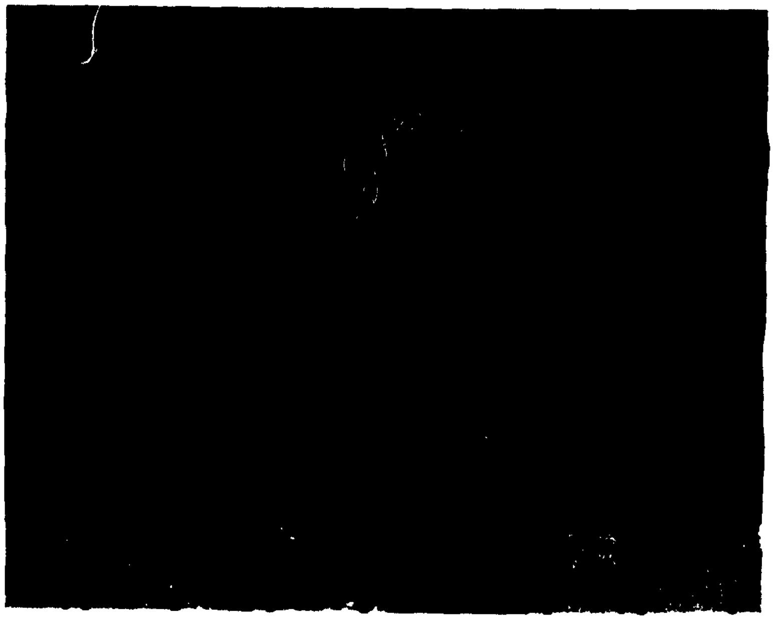
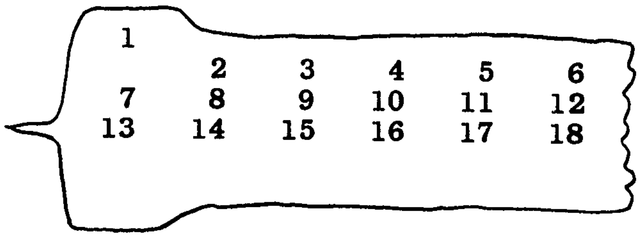


Figure 5: Macrostructure of Longitudinal and Transverse Sections of Forged and Annealed Cb74

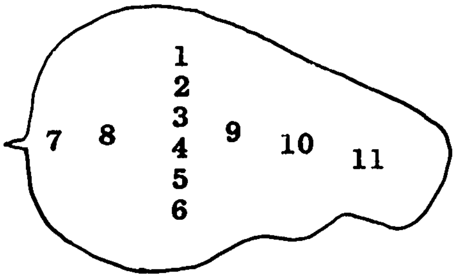
Figure 6

Vickers Hardness (10 Kg)



Longitudinal Section

	As Forged	Forged & Annealed 1 Hour at 2400F
1	225	209
2	248	201
3	225	188
4	233	231
5	225	229
6	279	224
7	234	215
8	218	224
9	220	197
10	234	226
11	229	217
12	221	222
13	233	230
14	233	217
15	231	226
16	240	235
17	212	201
18	226	245
Average	231	224



Transverse Section

	As Forged	Forged & Annealed 1 Hour at 2400F
1	233	235
2	230	230
3	234	229
4	224	224
5	237	229
6	234	227
7	252	226
8	241	225
9	261	222
10	241	226
11	229	229
Average	238	225

Figure 7

OXIDATION OF Cb74 (Cb-10W-4Zr)

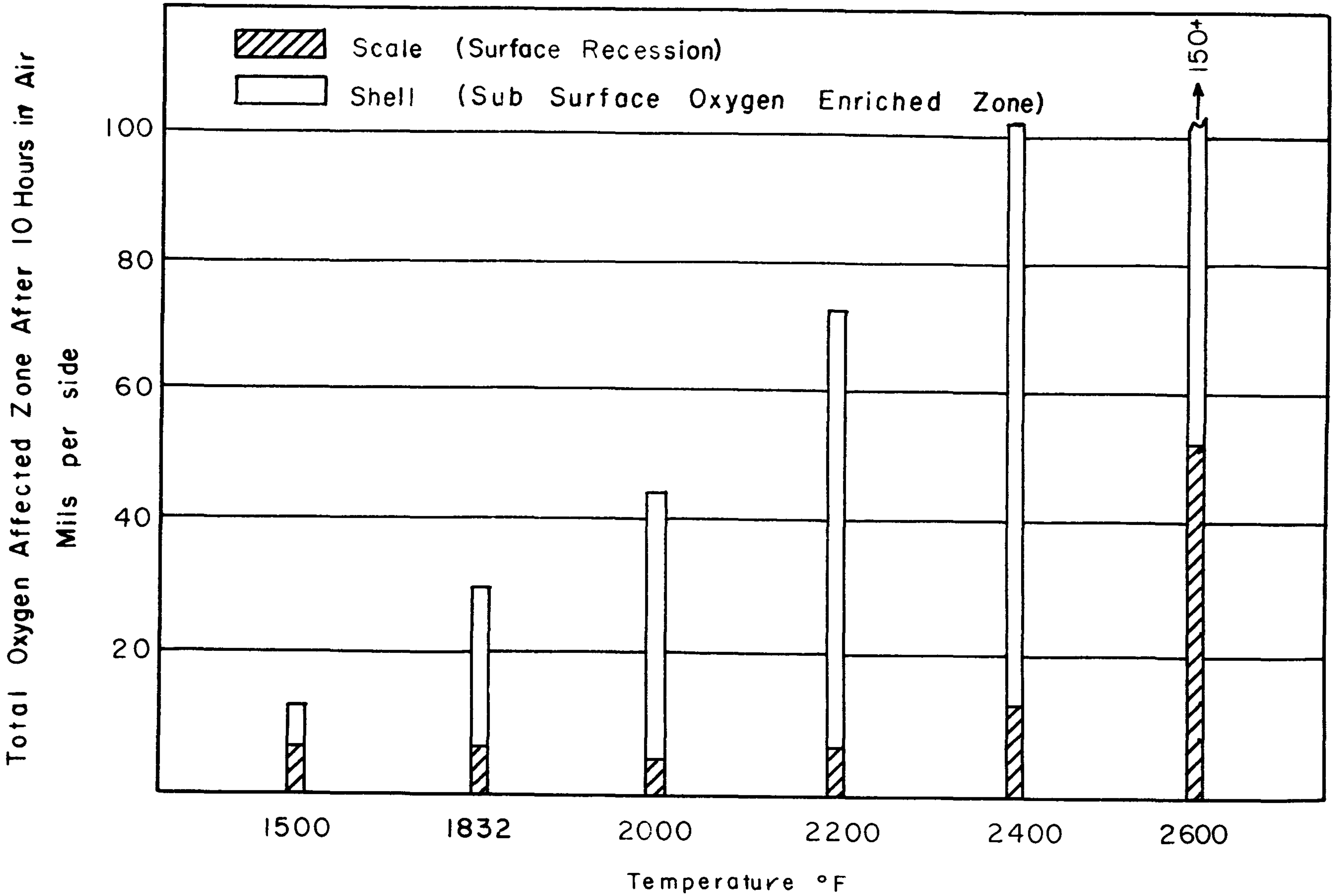


Figure 8: Microstructure of Cb74 Forging 719-4
Showing Slight Surface Contamination
300X

Figure 9

TENSILE STRENGTH OF Cb 74 (10W - 4 Zr)
(Annealed 1 Hour at 2400 °F)

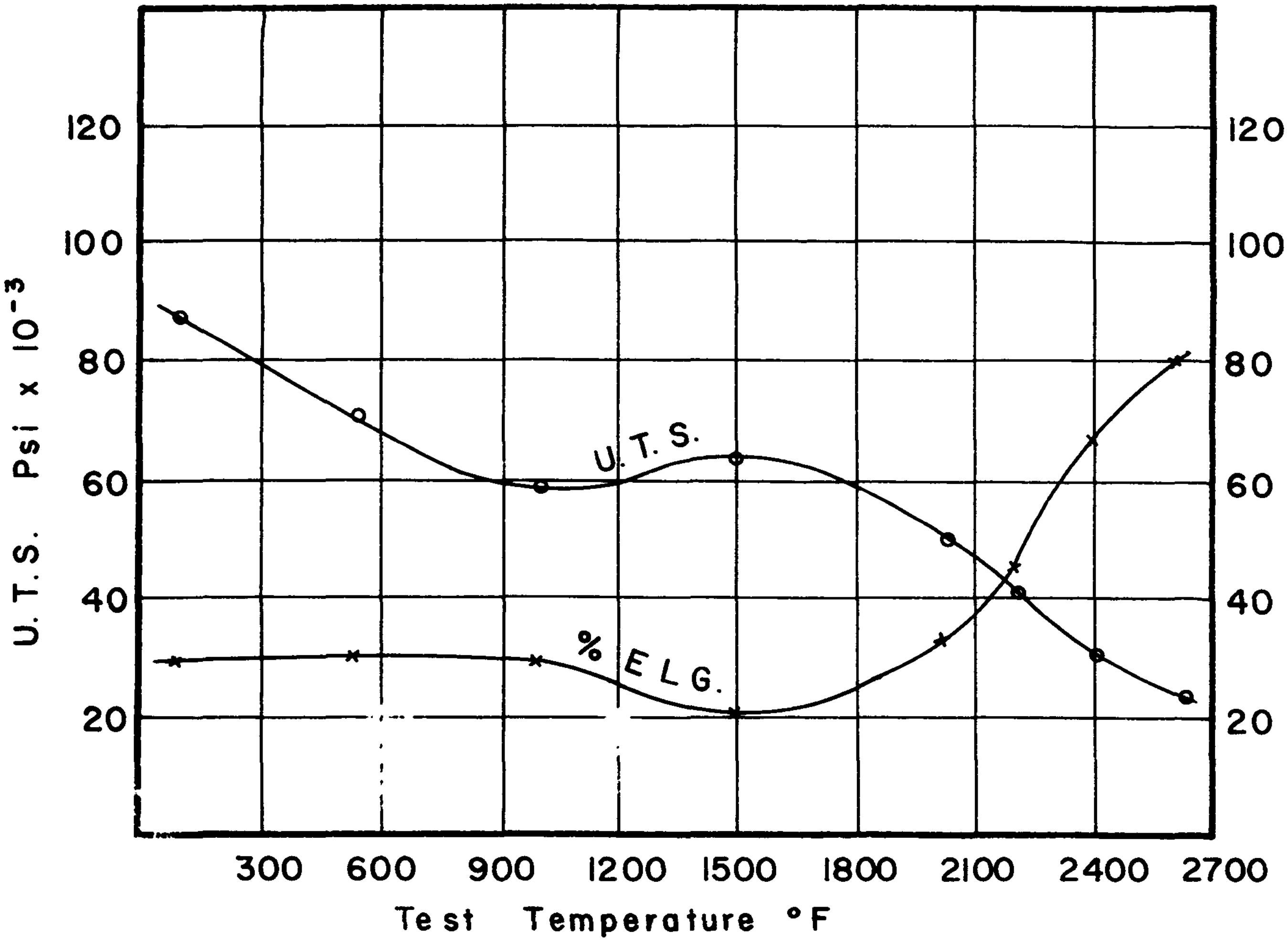
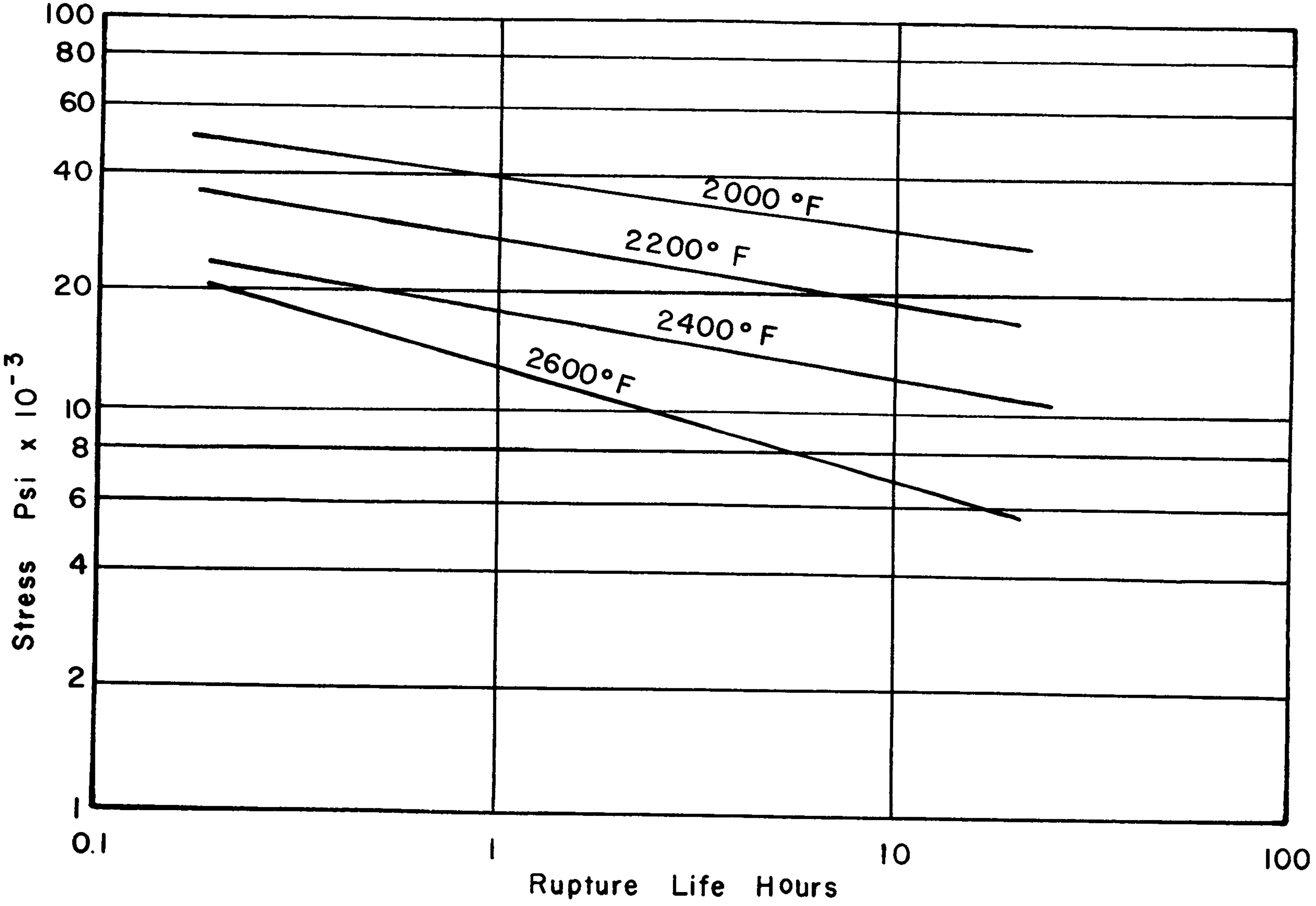


Figure 10

STRESS RUPTURE STRENGTH OF Cb 74



APPENDIX A

The following data which were obtained under Contract AF 33(600)-39942 "The Development of Optimum Manufacturing Methods for Columbium Alloy Sheet" completes the Ingot Integrity Evaluation and is the last phase of the columbium sheet contract that will be reported under this columbium forging contract.

1.0 D31 Ingot Homogeneity

A within-ingot and between-ingot uniformity study was made on the F48 alloy and was reported in the previous interim report. This uniformity study consisted primarily of chemical analyses from various ingot locations, structural examinations, and comparisons of mechanical properties and metallurgical characteristics of sheet processed in the laboratory from various ingot locations. A similar program has been conducted on the D31 alloy and is reported here.

Two 8" diameter D31 ingots (S98675 and S98676) were melted for this uniformity study by electron beam plus double vacuum arc remelt. This method of melting was found to be best for the D31 alloy by earlier work under this contract. Electron beam melting gives the required low interstitial content and vacuum arc remelting gives a more desirable ingot structure and permits the addition of titanium to the melt. Two vacuum arc remelts are necessary to obtain optimum titanium uniformity.

Top and bottom ingot slices and a longitudinal section through ingot S98676 are shown in Figures 1, 2 and 3. Structures are uniform and fine grained.

Analytical samples were taken from the ingot slices and are reported in Table I. In general, all elements are under good control and variability between ingot locations is not great. All molybdenum analyses except two are within the target range of 9.0 to 11.0%. The range of molybdenum results (1.8%) is less than the target range (2.0%) so a slightly altered formulation for future D31 ingots should bring all analyses within target limits. Titanium has a tendency to segregate to the ingot periphery; this confirms earlier findings under this contract. Due to this segregation, titanium results were lower than desired and varied over a wider range than molybdenum. Additional D31 melting experience is required to determine if the target range of 9.0 to 11.0% can be consistently met. Interstitial elements were under good control, though three carbon analyses on ingot S98675 were above the target range. Additional melting and testing experience will determine if this carbon content is detrimental and whether it can be controlled to the target range.

Five $2\frac{1}{4}$" diameter x 4" long billets were machined from ingot S98676 representing two ingot-top, one ingot-middle, and two ingot-bottom locations. These provide material for the within-ingot uniformity study. An oxidation-resistant coating was applied to all of the billets before forging to 1" slabs at 2350F. In addition to the coating, billets were encased in evacuated mild steel containers before forging. Forging technique was to side-forge 25%, turn 90°, and continue forging to final thickness. Two of the billets were forged in a hammer and three in a press for comparison of type of forging. On this test hammer forging appeared to be more satisfactory so ingot S98675 (8" diameter) was forged to 1-3/4" thick slab, at 2350F, by a hammer. The forging sequence was basically the same as that employed for the small billets--side forge 25%, turn 90°, and forge to 1-3/4" thick. An oxidation-resistant coating was applied to the ingot and it was vacuum sealed in a mild steel container before forging. Reheats were required during the forging of S98675. Since the mild steel can ruptured in the first few hammer blows, the ingot (including residual steel on its faces) was recoated to minimize oxidation and surface contamination during reheating. Intermediate conditioning at about 3" thick removed minor surface cracks which developed during initial forging. The ingot was recoated and recanned in mild steel before final forging to 1-3/4" thick.

In general, the above forging technique was satisfactory--surface contamination was minor, material losses were low, and the resulting slab product was suitable for rolling.

A piece of the S98675 forged slab was cut for laboratory processing to sheet along with the five forged billets from ingot S98676. Comparison of final properties provides the between-ingot uniformity study. All pieces from ingots S98675 and S98676 were processed to sheet in the laboratory by the cycle shown in Table II.

Mechanical property tests on the laboratory processed sheet are shown in Tables III to VII and are discussed below.

None of the tests (tensile tests at 0F, RT, 1000F, 1500F, 1800F, 2000F and 2200F, bend tests at RT and -110F, stress rupture tests at 2000F, and recrystallization tests) show significant variation between ingot positions or between ingots. Though less conclusive than a large body of test data on many ingots, this study is a reassuring indication that D31 alloy ingots by the selected melting process will yield uniform product.

The laboratory-processed D31 sheet is slightly directional, with higher strength and lower ductility in the transverse

direction than in the longitudinal. This difference persists up to about 1800F, where longitudinal and transverse strengths and ductility become about equal. D31 tensile elongation passes through a minimum at about 1000F to 1500F. This is similar to the behavior of F48 sheet and is probably due to a low rate of strain hardening rather than a lack of basic ductility. Room temperature tensile tests (Table IV) were also made on five of the D31 sheets 45° to the rolling direction because the tendency of columbium and its alloys to develop a strong (100) [110] rolling texture could result in different properties in this direction. However, strength is not significantly different from longitudinal and transverse test directions and ductility is slightly higher.

Bend tests (Table V) show that the D31 laboratory sheet has excellent minimum bend radii at room temperature and -110F. Bend transition, therefore, is below -110F.

As expected, stress rupture tests (Table VI) show that the D31 alloy is inferior to the F48 alloy (see Interim Report V, Tables III and VI) in elevated temperature creep resistance.

Table VII shows that recrystallization behavior is reasonably consistent between the various locations tested.

APPENDIX A

Table I

Chemical Analyses of Two 8" Diameter D31 Ingots (S98675 and S98676)

	Ingot S98675						Ingot S98676					
Ingot Position	% Mo	% Ti	% C	% N	% O	% H	% Mo	% Ti	% C	% N	% O	% H
Top Edge	9.6	10.8	.21	.007	.0110	.0007	8.9	7.6	.12	.012	.0098	.0004
Top Mid-Radius	9.7	10.3	.23	.009	.0114	0	9.2	8.1	.11	.014	.0074	.0005
Top Center	9.7	9.7	.22	.009	.0126	.0021	9.6	7.7	.12	.012	.0001	.0006
Middle Edge	-	-	-	-	-	-	9.4	6.7	.11	.018	.0114	.0002
Middle Center	-	-	-	-	-	-	8.4	7.3	.11	.012	.0061	.0008
Bottom Edge	9.4	7.8	.12	.009	.0100	.0004	10.2	8.2	.09	.008	.0105	.0007
Bottom Mid-Radius	9.4	8.0	.13	.008	.0083	.0002	10.2	8.1	.09	.009	.0085	.0005
Bottom Center	10.0	9.8	.21	.009	.0066	0	10.2	8.1	.09	.008	.0074	0

D31 target ranges -
Mo 9.0 - 11.0
Ti 9.0 - 11.0
C .08 - .12
N) Low as possible
O)

APPENDIX A

Table II

Laboratory Rolling Schedule for D31 Sheet

1. Coat and anneal one hour at 2500F.
2. Coat and hot roll to 6" x 0.400" x L at 2200F, recoating and reheating between each pass.
3. Coat and anneal one hour at 2200F.
4. Coat and hot roll to 6" x 0.100" x L at 2000F, recoating and reheating between each pass.
5. Coat and anneal one hour at 1750F.
6. Cold roll to 6" x 0.045" x L.
7. Coat and anneal one hour at 1750F.
8. Vacuum outgas at 1400F.

NOTE: Conditioning and pickling were used when necessary between rolling operations to remove surface defects and surface contamination. All sheets were finished with approximately 0.005" metal removal by pickling to remove residual contamination.

APPENDIX A

Table III

Longitudinal and Transverse Tensile Properties - 0.040" Laboratory Processed D31 Sheet

Test Temp	Heat Number	Ingot Location	Forging Technique	Longitudinal Ult Ten Str (ksi)	Longitudinal Yield Str (ksi)	Longitudinal Elong[2] (%)	Longitudinal Red in Area (%)	Transverse Ult Ten Str (ksi)	Transverse Yield Str (ksi)	Transverse Elong[2] (%)	Transverse Red in Area (%)
0F	S98676	Top 1	Press	116.9	(1)	23.3	54.2	121.0	(1)	23.3	45.2
		Top 2	Hammer	112.5	(1)	18.3	52.3	118.8	(1)	13.3	31.3
		Bottom 1	Press	109.7	(1)	38.3	41.9	107.1	(1)	10.0	34.9
		Bottom 2	Hammer	113.9	(1)	18.3	56.5	114.7	(1)	13.3	46.8
	S98675	-	Hammer	111.4	(1)	20.0	57.1	116.7	(1)	15.0	33.3
RT	S98676	Top 1	Press	109.1	97.8	14.5	42.4	107.3	98.7	8.0	35.4
				106.6	95.5	16.0	45.5	108.3	100.3	6.0	35.4
		Top 2	Hammer	103.8	92.6	16.5	48.1	108.5	94.5	8.5	28.6
				107.9	96.6	17.5	46.8	108.0	93.7	9.0	32.5
		Middle	Press	103.4	91.7	17.5	45.1	111.6	99.0	6.0	33.2
				102.7	91.8	14.0	48.5	111.0	99.4	4.0	33.8
		Bottom 1	Press	102.2	94.2	13.0	32.7	99.8	99.8	5.5	26.6
				102.0	92.5	11.5	35.1	100.3	94.7	4.0	24.0
		Bottom 2	Hammer	102.2	91.0	17.0	44.2	103.9	95.6	10.5	48.5
				103.7	89.6	18.5	40.3	103.4	95.1	10.0	51.0
	S98675	-	Hammer	102.4	91.9	10.0	45.2	107.9	98.6	8.5	37.6
				103.4	90.7	12.0	50.7	108.1	99.2	8.5	37.9
1000F	S98676	Top 1	Press	71.0	62.0	13.0	-	77.0	71.0	6.0	-
		Middle	Press	72.0	66.0	9.0	-	80.0	72.0	6.0	-
		Bottom 2	Hammer	71.0	63.0	9.0	-	75.0	69.0	6.0	-
	S98675	-	Hammer	67.0	59.0	9.0	-	76.0	68.0	6.0	-
1500F	S98676	Top 1	Press	64.0	54.0	14.0	-	68.0	57.0	3.0	-
		Middle	Press	64.0	53.0	13.0	-	72.0	62.0	6.0	-
		Bottom 2	Hammer	58.0	49.0	13.0	-	63.0	54.0	9.0	-
	S98675	-	Hammer	62.0	52.0	15.0	-	64.0	54.0	13.0	-

APPENDIX A

Table III
(Continued)

Test Temp	Heat Number	Ingot Location	Forging Technique	Longitudinal Ult Ten Str (ksi)	Longitudinal Yield Str (ksi)	Longitudinal Elong[2] (%)	Longitudinal Red in Area (%)	Transverse Ult Ten Str (ksi)	Transverse Yield Str (ksi)	Transverse Elong[2] (%)	Transverse Red in Area (%)
1800F	S98676	Top 1	Press	40.0	27.0	34.0	-	45.0	31.0	22.0	-
		Top 2	Hammer	41.0	27.0	33.0	-	45.0	35.0	18.0	-
		Middle	Press	41.0	27.0	38.0	-	44.0	32.0	25.0	-
		Bottom 1	Press	44.0	34.0	31.0	-	45.0	36.0	18.0	-
		Bottom 2	Hammer	39.0	28.0	41.0	-	44.0	31.0	25.0	-
	S98675	-	Hammer	37.0	26.0	39.0	-	44.0	31.0	37.0	-
2000F	S98676	Top 1	Press	28.0	18.0	41.0	-	28.0	19.0	45.0	-
		Top 2	Hammer	27.0	16.0	47.0	-	26.0	17.0	38.0	-
		Middle	Press	25.0	18.0	55.0	-	26.0	18.0	50.0	-
		Bottom 1	Press	19.0	14.0	72.0	-	23.0	18.0	47.0	-
		Bottom 2	Hammer	24.0	19.0	63.0	-	25.0	17.0	50.0	-
	S98675	-	Hammer	24.0	18.0	72.0	-	25.0	21.0	61.0	-
2200F	S98676	Top 1	Press	17.0	12.0	81.0	-	17.0	13.0	84.0	-
		Top 2	Hammer	16.0	12.0	88.0	-	15.0	11.0	72.0	-
		Middle	Press	15.0	12.0	84.0	-	17.0	13.0	86.0	-
		Bottom 1	Press	16.0	13.0	88.0	-	18.0	13.0	53.0	-
		Bottom 2	Hammer	15.0	12.0	91.0	-	16.0	12.0	84.0	-
	S98675	-	Hammer	15.0	11.0	62.0	-	16.0	12.0	95.0	-

(1) 0.2% offset yield strength could not be measured at 0F with equipment available.

(2) Measured over 2" gage length for room temperature tests and over 0.6" gage length for all other tests.

APPENDIX A

Table IV

Room Temperature Tensile Properties 45° to the Rolling Direction – 0.040" Laboratory Processed D31 Sheet

Heat Number	Ingot Location	Forging Technique	Ult Ten Strength (ksi)	Yield Strength (ksi)	Elongation (% in 2")	Reduction in Area (%)
S98676	Top 1	Press	96.0	93.1	17.5	52.2
	Top 2	Hammer	91.9	85.7	15.2	60.2
	Middle	Press	99.7	94.0	18.5	52.6
	Bottom 2	Hammer	96.5	91.5	19.0	52.6
S98675	–	Hammer	96.4	90.5	12.7	31.1

APPENDIX A

Table V

Room Temperature Bend Properties - 0.040" Laboratory Processed D31 Sheet

Heat Number	Ingot Location	Forging Technique	Room Temperature Minimum Transverse Bend Radius (x Thickness)	-110F Minimum Transverse Bend Radius (x Thickness)
S98676	Top 1	Press	1.6	2.0
	Top 2	Hammer	1.5	1.9
	Middle	Press	1.9	1.9
	Bottom 1	Press	1.9	1.9
	Bottom 2	Hammer	2.2	2.0
S98675	-	Hammer	1.5	1.9

APPENDIX A

Table VI

2000F Stress Rupture Data - 0.040" Laboratory Processed D31 Sheet

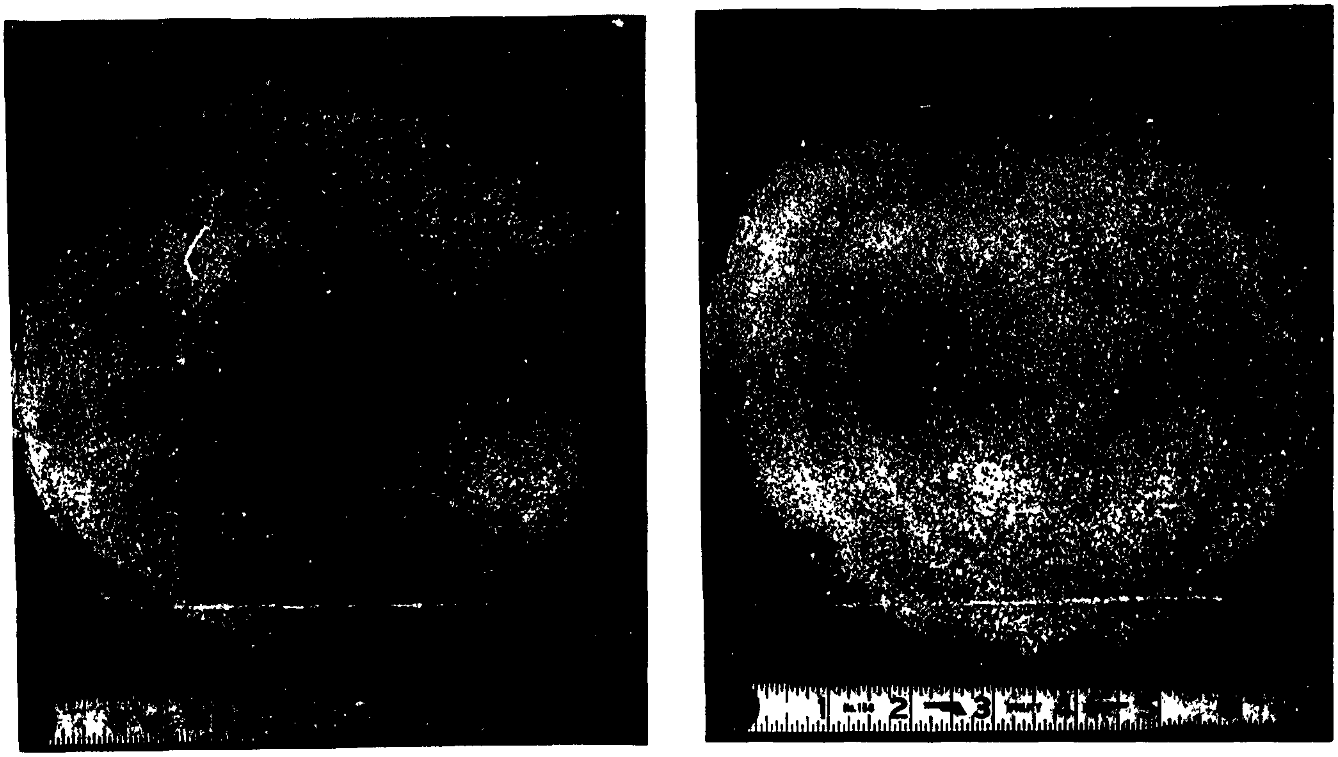
Heat Number	Ingot Location	Forging Technique	Stress (psi)	Life (Hours)	% Elongation
S98676	Top 2	Hammer	11,000	6.3	75
			11,000	19.0	84
	Bottom 1	Press	11,000	24.8	76
			11,000	21.8	95
	Bottom 2	Hammer	11,000	13.5	87
			11,000	16.6	62
S98675	-	Hammer	11,000	10.5	89
			17,500	0.6	67

APPENDIX A

Table VII

One-Hour Recrystallization Temperatures - 0.040" Laboratory Processed D31 Sheet Cold Rolled 50% And Stress Relieved One Hour at 1750F

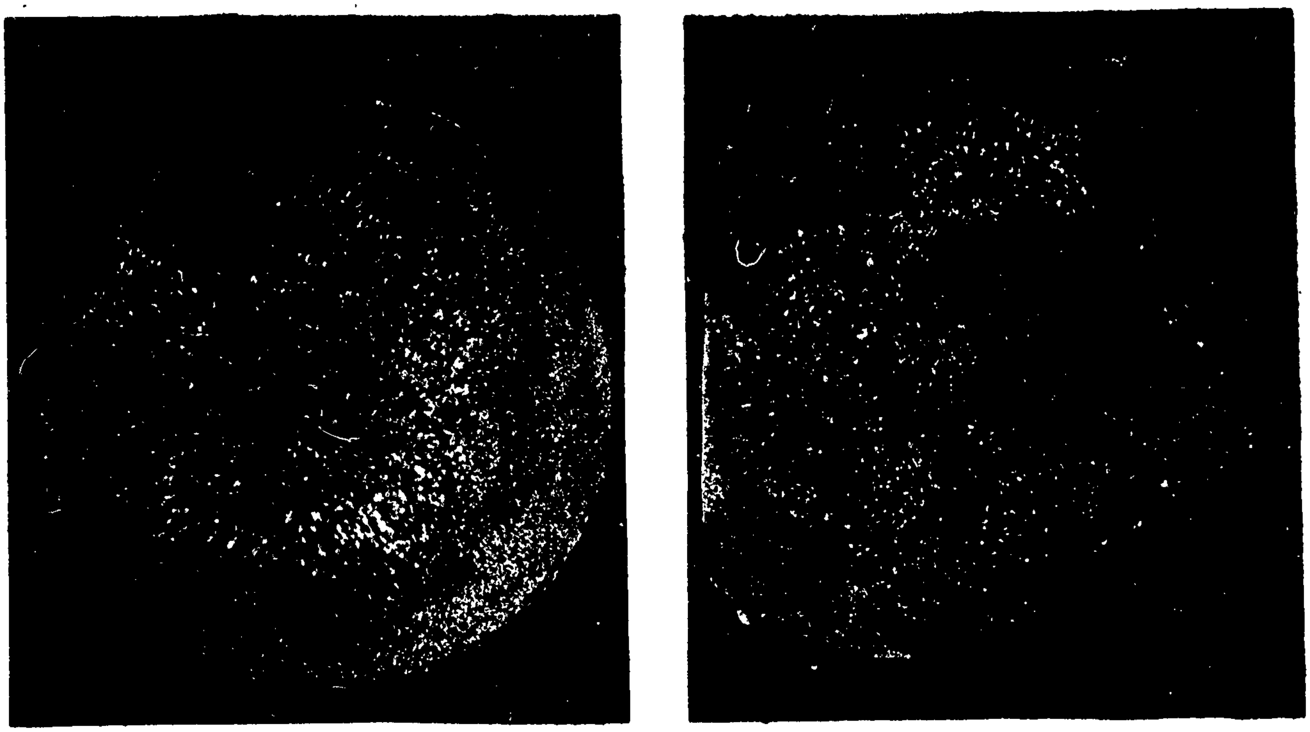
			Temperature For		
Heat Number	Ingot Location	Forging Technique	50% Recrys	75% Recrys	100% Recrys
S98676	Top 1	Press	1850F	1975F	2100F
	Top 2	Hammer	1850F	1975F	2100F
	Middle	Press	1800F	1925F	2100F
	Bottom 1	Press	1925F	2025F	>2100F
	Bottom 2	Hammer	<1800F	1875F	2000F
S98675	-	Hammer	1800F	1875F	2000F



Top

Bottom

Figure 1: Macrostructures of Top and Bottom Slices From D31 Ingot S98675. Titanium-rich skin on ingot surface can be seen.



Top   Bottom

Figure 2: Macrostructures of Top and Bottom Slices from D31 Ingot S98676. Titanium-rich skin on ingot surface can be seen. Magnification ½X

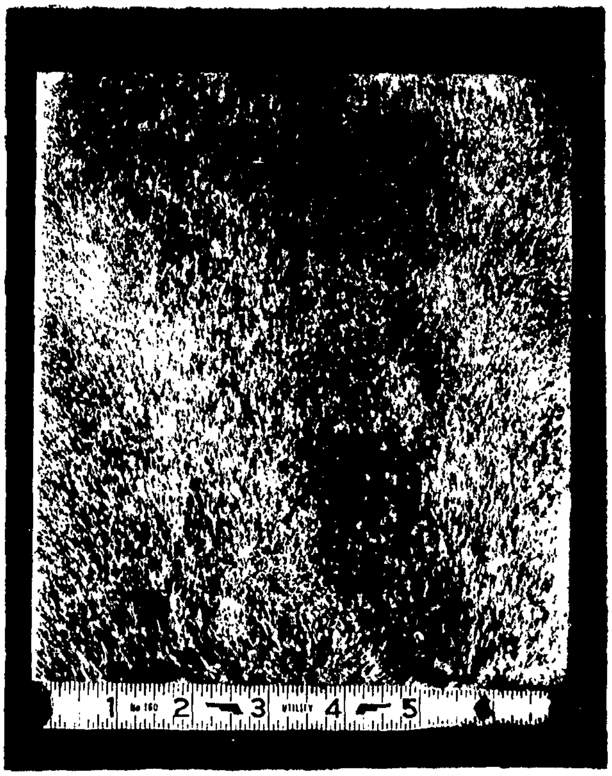


Figure 3: Macrostructure of Longitudinal Section Through D31 Ingot S98676.

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Sixteen Cb74 (10W-4Zr) closed die forgings were successfully made from 16 forging blanks
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